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LABORATORY B

REPORT NO.
KZ 1311

Report on

Tower Discharge Analysis

August 27, 1945

By J. A. Klacsmann

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Carbide and Carbon Chemicals Corporation, Operating Contractor for the U.S. Atomic Energy Commission.

Tower discharge samples were to be analyzed to estimate whether salvage would be economically advisable. Only the order of magnitude of material was needed.

The material came in a 3% basic Sodium carboriate solution, conatining approximately 10 mgs of material per liter.

The various methods tried were peroxide precipitation directly, sulfide precipitation directly, and an ammonia precipitation method (See Procedure I). These were each followed by a residue count. Besides these methods plating from the carboriate solution was tried, and the resultant film was counted.

The sulfide and peroxide methods gave unsatisfactory results, and further mention will not be given (See Table I).

The ammonia method worked satisfactorily, and a procedure was written (Procedure I).

Control samples were run as follows: A known amount of material of known counting rate was added to an unknown solution and the extra counts were determined experimentally. It was shown that 40% of the counts added were obtained.

Sample 1542 was run with 550 counts added to it as a control. 10 ml portion of 500 ml volume of 463 mg. of U_3O_8 (as nitrate) was diluted to 100 ml and 10 ml aliquot of that was added to sample (100 ml).

As can be seen from the data only 220 of the 550 counts added were obtained.

The answers were therefore multiplied by 2 1/2 before reporting (3 obtained by use of 1645).

The plating procedure (Procedure II) was worded out, and gave results comparable to the calculated Ammonia method.

Summary:

1. The results will be good the the desired certainty. (See Table I).
2. The plating method is a fast simple method which can be run by routine people.

TOWER DISCHARGE SAMPLES

Report on methods:

TABLE I

Sample No.	Method	Sample W and Mgs.	Volume ml.	Counts Per 100 ml.	
1542	NH_3	3.7	100	133	
1542 + 550 $\frac{Counts}{min}$	NH_3	5.5	100	350	
1564	NH_3	2.3	100	95	
1564 (Clear)	H_2S	49.9	100	11	
1564	Plating	___	30	240	
1585	NH_3	2.3	100	62	
1585	N_2S	49.6	100	17	
1594	NH_3	5.6	100	64	
1594	Plating	___	100	235	
1645	NH_3	5.9	100	64	
1645 + 550 $\frac{Counts}{min}$	NH_3	4.4	100	330	
1645	Plating(HNO_3)	___	100	35	
1786	Plating	___	100	73	
1786	Plating	___	100	71	69
1786	Plating	___	100	64	
1748	Plating	___	100	79	
1748	Plating	___	100	67	76
1748	Plating	___	100	81	

PROCEDURE I

PROCEDURE FOR NH_4OH PRECIPITATION ON DISPOSAL PLANT TOWER DISCHARGE

I. Acidify 100 ml of solution with nitric acid until solution shows acid.

II. Evaporate acidified solution to dryness.

III. Add 10 ml. concentrated nitric acid, washing down sides of beaker with acid.

IV. Evaporate until dry and bake until residue stops omitting acid fumes.

V. Dissolve the reside in a minimum of C_{o_2} free water.

VI. Heat solution just to boiling and add an equal volume of Co_2 free 4 M NH_4OH.

VII. Centrifuge for 30 seconds.

VIII. Pour off excess liquid.

IX. Add concentrated nitric acid dropwise until precipitate dissolves being careful not to use an excess of acid.

X. Add 4 M NH_4OH in amount equal to solution of sample.

XI. Centrifuge for 30 seconds and pour off excess liquid.

XII. Dry precipitate in centrifuge tube under infara - rediater until the ppt. becomes hard.

XIII. Loosen the dried precipitate with a platinum spotula and transfer to a weighted platinum dish.

XIV. Ignite sample for 15 minutes and weigh.

XV. Dissolve ignited sample in nitric acid.

XVI. Evaporate solution to dryness.

XVII. Heat sample gently until it turns to a red color.

XVIII. Add 4 M HCL to dissolve.

XIX. Transfer sample to silver foil disc for correcting as follows:

a. Place disc in bottom of petri dish placed on a hot plate.
b. Drop sample solution on disc from a dropper no faster than the solution will evaporate to form a deposit in the middle portion of the disc.
c. Rinse platinum dish twice with some 4 M HCL.
d. Ignite disc in oven for one minute at H 25° C.